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*Indian Standard*

METHODS OF ESTIMATION OF  
COMPOSITE PIGMENTS IN OIL PASTES, READY  
MIXED PAINTS AND ENAMELS

PART II ESTIMATION OF ZINC CHROMES, FERRIC  
OXIDE AND ALUMINIUM

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*Indian Standard*METHODS OF ESTIMATION OF  
COMPOSITE PIGMENTS IN OIL PASTES, READY  
MIXED PAINTS AND ENAMELSPART II ESTIMATION OF ZINC CHROMES, FERRIC  
OXIDE AND ALUMINIUM

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# *Indian Standard*

## METHODS OF ESTIMATION OF COMPOSITE PIGMENTS IN OIL PASTES, READY MIXED PAINTS AND ENAMELS

### PART II ESTIMATION OF ZINC CHROMES, FERRIC OXIDE AND ALUMINIUM

#### 0. FOREWORD

**0.1** This Indian Standard ( Part II ) was adopted by the Indian Standards Institution on 25 February 1975, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

**0.2** While finalizing IS : 101-1964\* for publication, the concerned Sectional Committee was confronted with the problem whether to include standard methods of tests for single pigments or not. It was decided that under 27.1.1.1 of IS : 101-1964\* it may be clearly indicated that analysis of single pigments shall be done as specified in individual specifications and analysis of composite pigments in accordance with a separate standard. Consequently, formulation of this standard was taken up. The Committee, however, agreed to issue the standard in parts. Consequently IS : 6947( Part I )-1973† was first formulated. This standard ( Part II ) covers methods of estimation of zinc chromes, ferric oxide and aluminium. The third and final part in the series is covered by IS : 6947 ( Part III )-1975‡.

**0.3** This standard is a necessary adjunct to material specifications for oil pastes, ready mixed paints and enamels.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960§.

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\*Methods of test for ready mixed paints and enamels ( *second revision* ).

†Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels: Part I Estimation of lead, zinc oxide, titanium dioxide, calcium compounds and zinc sulphide.

‡Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels: Part III Estimation of lead chromes, iron blues and zinc compounds.

§Rules for rounding off numerical values ( *revised* ).

# **IS : 6947 ( Part II ) - 1975**

## **1. SCOPE**

**1.1** This standard ( Part II ) prescribes methods of estimation of zinc chromes, ferric oxide and aluminium in composite pigments of oil pastes, ready mixed paints and enamels.

## **2. TERMINOLOGY**

**2.1** For the purpose of this standard, the definitions given in 2 of IS : 101-1964\* and IS : 1303-1963† shall apply.

## **3. QUALITY OF REAGENTS**

**3.1** Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070-1960‡ ) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

## **4. EXTRACTION OF PIGMENTS**

**4.1** Pigments from oil pastes, ready mixed paints and enamels shall be first extracted in accordance with the procedure given in 27 of IS : 101-1964\*.

## **5. DETERMINATION OF CHROMIC ANHYDRIDE AND ZINC OXIDE**

**5.0 Outline of the Method** — Chromic anhydride is determined by titrating the iodine liberated from potassium iodide with standard sodium thiosulphate solution. Zinc oxide is determined in the same solution by titrating the iodine subsequently liberated on addition of potassium ferricyanide, with standard sodium thiosulphate solution.

**5.0.1** Two methods are prescribed. Method A is followed where iron compounds are absent and Method B is followed for estimation in presence of iron compounds.

### **5.1 Reagents**

**5.1.1** *Dilute Sulphuric Acid* — approximately 7 N.

**5.1.2** *Potassium Iodide Solution ( freshly prepared )* — 10 percent.

**5.1.3** *Standard Sodium Thiosulphate Solution* — 0.1 N.

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\*Methods of test for ready mixed paints and enamels ( second revision ).

†Glossary of terms relating to paints ( revised ).

‡Specification for water, distilled quality ( revised ).



**5.1.4 Starch Solution** — 0.5 percent.

**5.1.5 Ammonia Solution** — 1 : 1 ( v/v ).

**5.1.6 Potassium Ferricyanide Solution** — M/10 prepared in cold distilled water, filtered rapidly and kept in a dark bottle. The solution can be suitably used for seven days if kept in dark when not in use.

**5.1.7 Ammonium Bifluoride** (  $\text{NH}_4$  )  $\text{HF}_2$  — solid.

**5.1.8 Sodium Hydroxide Solution** — 10 percent ( approximately ).

## 5.2 Method A

### 5.2.1 Procedure

**5.2.1.1** Weigh accurately about 0.5 g of the extracted pigment in a 500-ml conical flask. Add 70 ml of water and 30 ml of dilute sulphuric acid. Boil slowly for 2-3 minutes and cool.

**5.2.1.2** Transfer the solution to a 500-ml iodine flask containing 100 ml water and 20 ml dilute sulphuric acid. Add 30 ml of potassium iodide solution and allow to stand for 5 minutes. Titrate the liberated iodine with standard sodium thiosulphate solution after adding 5 ml of starch solution as indicator. Note the volume of sodium thiosulphate solution used in the titration (  $V_1$  ).

**5.2.1.3** After the titration for chromic anhydride, add ammonia solution dropwise until the litmus test paper first turns blue. ( An excess of ammonia solution, more than 3 to 4 drops, should not be added as it is essential that the blue litmus should turn red with ammonium bifluoride to be added subsequently. ) Cool and add 2 to 3 g of ammonium bifluoride and sufficient water to make volume approximately 250 ml. ( Sometime traces of iodine liberated during neutralization can be cleared up by one or two drops of sodium thiosulphate solution. ) Add  $V_1/3$  ml of potassium ferricyanide solution. Stir and allow to stand for about 2 minutes. Titrate the liberated iodine with standard sodium thiosulphate solution. After the end point the colour of the test solution will be bright pale-greenish yellow. Note the volume of sodium thiosulphate solution used in the second titration (  $V_2$  ). In case  $V_2$  is less than 6 ml, the titration is to be repeated with the addition of (  $V_2 + 2$  ) ml of potassium ferricyanide solution in place of  $V_1/3$  ml.

**5.2.1.4** If  $V_2$  is less than 3 ml, the titration is to be repeated, starting from extracted pigment, with the addition of 5 ml potassium ferricyanide solution.

**5.2.2 Calculation**

a) Chromic anhydride (  $\text{CrO}_3$  ),  
percent by mass 
$$= \frac{3.334 \times V_1 \times N}{M}$$

where

$V_1$  = volume in ml of sodium thiosulphate solution used in first titration,

$N$  = normality of sodium thiosulphate solution used, and

$M$  = mass in g of the material taken for the titration.

b) Zinc oxide (  $\text{ZnO}$  ),  
percent by mass 
$$= \frac{12.45 \times V_2 \times N}{M}$$

where

$V_2$  = volume in ml of sodium thiosulphate solution used in the second titration, and

$N$  and  $M$  have the same legend as in (a) above.

**5.3 Method B****5.3.1 Procedure**

**5.3.1.1** Weigh accurately about 1 g of the pigment in a 250-ml beaker and add about 100 ml of sodium hydroxide solution. Stir well by rubbing with a glass rod and boil slowly for five minutes over a wire gauze. Place the beaker, covered with a clock glass on a water-bath for about half an hour. Filter over filter paper ( Whatman No. 42 ) in a 500-ml conical flask. Wash well with hot water until the residue is free from chromate. Cool the filtrate in ice water and neutralize it with dilute sulphuric acid. Add 30 ml of dilute sulphuric acid after neutralization and cool. Then estimate chromic anhydride and zinc oxide content as given in **5.2.1.2** to **5.2.1.4**.

**5.3.1.2** If  $V_2$  is less than 2 ml, the titration is to be repeated, starting from the extracted pigment, with the addition of 5 ml of potassium ferricyanide solution.

**5.3.2 Calculation**

a) Chromic anhydride (  $\text{CrO}_3$  ),  
percent by mass 
$$= \frac{3.334 \times V_1 \times N}{M}$$

where

$V_1$  = volume in ml of sodium thiosulphate solution used in the first titration,

$N$  = normality of sodium thiosulphate solution used, and

$M$  = mass in g of material taken for the titration.

$$\text{b) Zinc oxide ( ZnO ),} \\ \text{percent by mass} = \frac{12.45 \times V_2 \times N}{M}$$

where

$V_2$  = volume in ml of sodium thiosulphate solution used in the second titration, and

$N$  and  $M$  have the same legend as in (a) above.

## 6. ESTIMATION OF RED OXIDE OF IRON

**6.0 Outline of the Method** — The pigment is dissolved in hydrochloric acid and the ferric ion reduced to ferrous state with stannous chloride solution. Ferric oxide is estimated by oxidimetric titration of ferrous solution using standard solution of potassium dichromate.

### 6.1 Reagents

**6.1.1 Standard Potassium Dichromate Solution (0.1N)** — Finely powder about 6 g of potassium dichromate in a glass or agate mortar. Heat for about an hour in an air oven at 140 to 150°C. Cool in a desiccator. Dissolve 4.904 g of the accurately weighed powder in water in a one-litre measuring flask. Shake thoroughly and make up to the mark.

**6.1.2 Stannous Chloride Solution** — Dissolve 12 g of pure tin or 30 g of crystallized stannous chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) in 100 ml of hydrochloric acid (relative density 1.16) and dilute with water to 200 ml.

**6.1.3 Hydrochloric Acid** — Relative density 1.16 ( see IS : 265-1962\* ).

**6.1.4 Dilute Sulphuric Acid** — 2.5 percent by volume.

**6.1.5 Phosphoric Acid** — 85 percent by volume.

**6.1.6 Saturated Aqueous Solution of Mercuric Chloride**

**6.1.7 Diphenylamine Indicator** — 1 percent solution in concentrated sulphuric acid.

**6.2 Procedure** — Weigh accurately about 0.3 g of the extracted pigment. Take it in a 400-ml beaker and add about 50 ml of dilute hydrochloric acid. Cover the beaker with a clock glass and place it on the water-bath for about 4 hours ( or overnight ). Then gently boil the contents of the beaker over a wire gauze for a few minutes to get the pigment ( excepting siliceous matter ) into complete solution. Filter over filter paper ( Whatman No. 30 ) into a 500-ml conical flask, wash with hot water and evaporate the solution to about 50 ml. Heat to boiling and add the solution of stannous chloride dropwise, with agitation after each addition,

\*Specification for hydrochloric acid ( revised ).

until the liquid is colourless or has a faint green colour, quite free from any tinge of yellow. Add two to three drops in excess, cool the solution rapidly, add about 20 to 25 ml of saturated solution of mercuric chloride and allow it to stand for 3 to 5 minutes when a slight silky white precipitate is obtained. Then add about 200 ml of dilute sulphuric acid, 5 ml of phosphoric acid and 3 to 4 drops of diphenylamine indicator. Titrate slowly and with constant stirring against standard potassium dichromate solution to the first permanent violet blue colouration.

**6.3 Calculation** — Calculate the amount of ferric oxide, taking 1 ml of 0.1 N potassium dichromate equivalent to 0.008 g of ferric oxide ( $\text{Fe}_2\text{O}_3$ ) as given below:

$$\text{Ferric oxide (Fe}_2\text{O}_3\text{), percent by mass} = \frac{8 \times V \times N}{M}$$

where

$V$  = volume in ml of potassium dichromate solution used,

$N$  = normality of potassium dichromate solution used, and

$M$  = mass in g of pigment taken for the test.

## 7. ESTIMATION OF ALUMINIUM PIGMENTS

**7.0 Outline of the Method** — The pigment (after making it grease-free) is treated with sodium hydroxide solution, filtered and made slightly acidic with hydrochloric acid. Then aluminium is precipitated as the hydrated oxide by means of ammonia solution. The precipitate is washed, converted into the oxide by ignition and weighed as  $\text{Al}_2\text{O}_3$ .

### 7.1 Reagents

**7.1.1 Sodium Hydroxide Solution** — 10 percent.

**7.1.2 Hydrochloric Acid Solution** — 1 : 1.

**7.1.3 Ammonia Solution** — 1 : 1.

**7.1.4 Ammonium Chloride** — pure.

**7.1.5 Methyl Red Indicator** — 0.2 percent alcoholic solution.

**7.1.6 Ammonium Nitrate Solution** — 2 percent.

**7.2 Procedure** — Weigh accurately a suitable quantity of the extracted grease-free pigment (containing approximately 0.1 to 0.2 g of aluminium) and dissolve it in 50 ml of sodium hydroxide solution in a 250-ml beaker provided with a clock glass cover. When vigorous reaction subsides, heat on the water-bath till the reaction is complete. Dilute with 100 ml of hot

water, filter through a sintered glass crucible ( No. 4 ), and wash thoroughly with hot water. Take the filtrate in a 400-ml beaker and add hydrochloric acid solution till the precipitate formed during neutralization practically redissolves. Add 5 g of ammonium chloride, sufficient water to make volume approximately 250 ml and a few drops of methyl red indicator. Heat just to boiling, add ammonia solution dropwise till the colour of the solution changes to yellow and boil for 1 to 2 minutes. Filter in about 15 minutes through a filter paper ( Whatman No. 31 ). Transfer the filter paper with the precipitate to the same beaker, pierce the filter paper with a glass rod, dissolve the precipitate in about 10 to 15 ml of hydrochloric acid solution and add 200 ml of water. Reprecipitate after adding 2 to 3 g of ammonium chloride and a few drops of the indicator as above. Wash the precipitate 6 to 7 times with hot ammonium nitrate solution. Place the paper with the precipitate in a previously ignited weighed platinum crucible, dry, char and ignite for about 15 minutes ( at a temperature of about  $1200^{\circ}\text{C}$  ) in a muffle furnace. Cool the crucible in a desiccator and weigh. Ignite to constant mass.

### 7.3 Calculation

$$\text{Aluminium, percent by mass} = \frac{M_1 \times 52.91}{M}$$

where

$M_1$  = mass in g of the ignited alumina (  $\text{Al}_2\text{O}_3$  ), and

$M$  = mass in g of the extracted pigment taken for analysis.

( Continued from page 2 )

**Panel for Organic Pigments, CDC 50 : 1 : 2**

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IS:

- 85-1950 Methods of test for oil pastes for paints
- 101-1964 Methods of test for ready mixed paints and enamels (*second revision*)
- 6947 (Part I)-1973 Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels: Part I Estimation of lead, zinc oxide, titanium dioxide, calcium compounds and zinc sulphide
- 6947 (Part II)-1975 Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels: Part II Estimation of zinc chromes, ferric oxide and aluminium
- 6947 (Part III)-1975 Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels: Part III Estimation of lead chromes, iron blues and zinc compounds

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